Analysis of Perchlorate in Lettuce

The Food and Drug Laboratory (FDL) of the Department of Health Services has modified the method for determining perchlorate in lettuce developed by Ellington and Evans (Journal of Chromatography A, 2000, 898: 193-199). The purpose of the modification was to accommodate for the existing equipment in FDL and to lower the method's perchlorate detection limit. FDL achieved a lower detection limit by reducing the amount of water for extraction and dilution. FDL's modified method is described below. For more detailed information, please either refer to the published article of Ellington and Evans[1] or contact the Food and Drug Branch (FDB) at (916) 445-2264.

Modified FDL Method

1. Lettuce Preparation

The lettuce used for the modification at FDL was purchased from a local supermarket. The lettuce was cored and the leaves cut into 1-2 cm pieces. It was oven-dried* at 50^{o}C for about 16-18 hours. The dried lettuce was weighed and its weight was recorded. Then, the dried lettuce was pulverized using a mortar and pestle.

2. Sample Preparation

Six hundred mg of pulverized lettuce was weighed and placed in a 50 mL centrifuge tube. Twenty mL of deionized water* were added to the centrifuge tube. The tube was tightly capped and vortexed for 3 minutes. The tube was placed in a boiling water bath for 30 minutes. Then, the tube was placed in the refrigerator overnight. Total time of extraction was 20 hours. The tube was removed from refrigerator and vortexed for 1 minute* and centrifuged at 4,000 rpm for 30 minutes*. The supernatant was decanted into a 50 mL vial. Approximately 5 mL of the supernatant was taken from the vial and filtered with a 0.45 μm Acrodisc^{R} syringe filter*. One gram of activated alumina was weighed and placed in a 20 mL vial, and 2 mL of the filtered supernatant was added to the vial containing the activated alumina. The vial was let stand in the refrigerator overnight. Then, 8 mL of deionized water was added to the vial (1/5 dilution)* and mixed thoroughly. The vial was let stand for 30 minutes. The solution in the vial was filtered using an activated On Guard^{R} RP cartridge and a second 0.45 μm Acrodisc^{R} syringe filter in tandem. The first 3 mL of the filtrate was discarded and the rest filtrate was collected into a 5 mL auto-sampler vial for the Ion Chromatography analysis of perchlorate.

3. Instrumentation

Ion chromatography was performed on a Dionex DX-500 system. The chromatograph was equipped with Isocratic pump (IP25)*, Eluent Generator (EG40)*, and CD20 conductivity detector. An ASRS^{R}ULTRA (4 mm*) suppressor was used. A guard column AG 16 (4 x 50 mm)* and an analytical column AS 16 (4 x 250 mm) were used

for the separation of anions. Samples were run in isocratic mode using 65 mM KOH* as eluent. The system was run in recycle mode, and the sample injection volume was 1 mL.

4. Other equipment and materials

Refer to those specified in the published article of Ellington and Evans[1].

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* Different from that of the method of Ellington and Evans[1].

1. J. Jackson Ellington and John J. Evans, 2000, Journal of Chromatography A, 898: 193-199

To download: visit http://www.epa.gov/athens/research/regsupport/index.html and click "Ellington and Evans' method for the analysis of perchlorate (PDF Format)."

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